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## IGNITION AND COMBUSTION OF BULK METALS IN A MICROGRAVITY ENVIRONMENT

M. C. BRANCH, J. W. DAILY, AND A. ABBUD-MADRID  
CENTER FOR COMBUSTION RESEARCH  
MECHANICAL ENGINEERING DEPARTMENT  
UNIVERSITY OF COLORADO  
BOULDER, CO 80309-0427

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## ABSTRACT

This annual report summarizes the latest results obtained in a NASA-supported project to investigate the effect of gravity on the ignition and combustion of bulk metals. The experimental arrangement used for this purpose consists of a 1000-W xenon lamp that irradiates the top surface of cylindrical titanium and magnesium specimens, 4 mm in diameter and 4 mm in height, in a quiescent, pure-oxygen environment at 1 atm. Reduced gravity is obtained from the NASA-LeRC DC-9 aircraft flying parabolic trajectories. Values of critical and ignition temperatures are obtained from thermocouple records. Qualitative observations and propagation rates are extracted from high-speed cinematography. Emission spectra of gas-phase reactions are obtained with an imaging spectrograph/diode array system. It was found that high applied heating rates and large internal conduction losses generate critical and ignition temperatures that are several hundred degrees above the values obtained from isothermal experiments. Because of high conduction and radiation heat losses, no appreciable effect on ignition temperatures with reduced convection in low gravity is detected. Lower propagation rates of the molten interface on titanium and of ignition waves on magnesium are obtained at reduced gravity. These rates are compared to theoretical results from heat conduction analyses with a diffusion/convection-controlled reaction. The close agreement found between experimental and theoretical values indicates the importance of the influence of natural convection-enhanced oxygen transport on combustion rates. Lower oxygen flux and lack of oxide product removal in the absence of convective currents appear to be responsible for longer burning times of magnesium diffusion flames at reduced gravity. The accumulation of condensed oxide particles in the flame front at low gravity produces a previously unreported unsteady explosion phenomenon in bulk magnesium flames. This spherically symmetric explosion phenomenon seems to be driven by increased radiation heat transfer from the flame front to an evaporating metal core covered by a porous, flexible oxide coating. These important results have revealed the significant role of gravity on the burning of metals, and are now being used as the database for future experiments to be conducted with different metals at various pressures, oxygen concentrations and gravity levels.

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## I. SUMMARY OF THE OBJECTIVE AND VALUE OF THIS RESEARCH

For the past five years, metal combustion studies at the University of Colorado have focused on the effects of gravity ( $g$ ) on the ignition and burning behavior of bulk metals. The impetus behind this effort is the understanding of the ignition conditions and flammability properties of structural metals found in oxygen ( $O_2$ ) systems for space applications. Since spacecraft are subjected to higher-than-1  $g$  loads during launch and reentry and to a zero-gravity environment while in orbit, the study of ignition and combustion of bulk metals at different gravitational accelerations is of great practical concern. From the scientific standpoint, studies conducted under low gravity conditions provide simplified boundary conditions, since buoyancy is removed, and make possible the identification of fundamental ignition and combustion mechanisms.

The influence of microgravity on the burning of bulk metals has previously been investigated by Steinberg et al. [1] in a drop tower simulator. All metals and alloys tested supported combustion in the absence of gravity. In general, the study found that in microgravity the upward regression rate of the melting surface of a metal rod is significantly faster than in normal gravity. From a review of some of the theoretical models of bulk metal combustion [2-4], it is believed that gravity plays an important role in heat and mass transport processes that dominate metal-oxygen reactions. The possible significance of convection on mass transfer rates and on the outward transport of condensed oxides in vapor-phase metal diffusion flames was first mentioned by Brzustowski and Glassman [5].

The present investigation is intended to provide experimental verification of the influence of natural convection on the burning behavior of metals. In addition, the study offers the first findings of the influence of gravity on ignition of bulk metals and on the combustion mechanism and structure of metal-oxygen, vapor-phase diffusion flames. The research work done to date has involved the use of several facilities to study the ignition and combustion phenomena under various gravity conditions. The last set of experiments have consisted of reduced gravity tests conducted onboard the NASA-LeRC DC-9 aircraft capable of flying parabolic trajectories that provide  $10^{-2}$   $g$  levels. These experiments were recently performed during two one-week sets of missions. This report summarizes the main results obtained from the normal and reduced gravity tests.

## II. EXPERIMENTAL SYSTEM

Titanium (Ti) and magnesium (Mg) metals were selected as the test samples because of their importance as elements of structural materials and their simple chemical composition—pure metals instead of multicomponent alloys to simplify chemical and spectroscopic analyses. In addition, these elements present the two different combustion modes observed in metals: heterogeneous or surface burning (Ti) and homogeneous or gas-phase reaction (Mg). Finally, Mg, Ti, and their oxides exhibit a wide range of thermophysical and chemical properties.

A schematic of the experimental system used is shown in Fig. 1. The ignition source consists of a 1000-W xenon lamp that generates a highly collimated beam ( $4^\circ$  half angle) with broadband radiation (300-1100 nm). An aspheric lens focuses the beam to provide a  $1.75\text{-MW/m}^2$  power density on the top surface of a 4-mm-diameter and 4-mm-high metal specimen that

sits on an alumina holder. An electric shutter permits effective control of heating time. A 4.5-L, stainless steel, cylindrical combustion vessel houses the lens, metal specimen, and alumina holder. Optical access for the movie camera and spectrograph is provided through two fused-silica side windows, while a third window is used for sample replacement. Ti and Mg metal specimens (99.95% purity) are placed in an oxygen environment (99.6% min.) at an absolute pressure of 1 atm. Evacuation and filling of the vessel is accomplished with a series of computer-controlled solenoid valves. The chamber pressure is monitored with a solid-state piezoresistive transducer. The surface temperature is measured with a 0.125-mm diameter, Type *R* thermocouple attached to the outer wall of the sample. A second thermocouple is sometimes used in different locations to measure temperature gradients within the sample.

A high-speed, 16-mm movie camera provides surface and flame visualization; the images are also used for measurement of propagation rates. With a 7.5° shutter and speeds up to 500 frames/s, exposure times as short as 1/20,000 s are obtained. A 50-mm lens and various extension tubes are used for image magnification. In addition to visible light imaging, time- and space-resolved spectral information on gas-phase reactants and products is obtained with an imaging spectrograph and a 1024-element diode array detector. Various spectral ranges are covered with two motorized gratings (300 and 2400 grooves/mm). The output signal from the detector is processed by an external controller that delivers a 15-bit dynamic range and a 150-Hz readout rate with direct memory access.

The experiment is controlled entirely by a computer, a digital/analog data acquisition board, and an interface code written in graphical programming software. In a typical run, the complete sample assembly (clean metal specimen mounted in alumina holder with attached thermocouple) is introduced into the chamber. Five evacuation and filling cycles are executed to provide a pure-oxygen atmosphere inside the vessel. The lamp is adjusted to the desired output power and the computer signals the shutter to open when the target gravity level (measured from a three-axis accelerometer located near the chamber) is achieved. Data acquisition starts (with 1-ms resolution), and the signal from the thermocouple is used to trigger all events. Immediately after ignition, the shutter is closed to remove all external heating to the sample. Temperature, pressure, energy input, gravity level, high-speed photography, and emission spectra are monitored throughout the experiment. After complete combustion, the final pressure is recorded to quantify oxygen consumption, and the burned or quenched samples are stored for chemical composition studies.

### III. LOW-GRAVITY FLIGHT EXPERIMENTS

The low-gravity experiments were conducted onboard the NASA-Lewis DC-9 Research Aircraft in Cleveland, Ohio during two one-week sets of missions (September 11-15 and December 4-8, 1995). Up to 20 s of reduced gravity ( $\pm 0.01 g$ ) were available in a single parabolic maneuver. Ten tests were conducted for each metal at reduced gravity to ensure experimental repeatability.

#### IV. HEATING AND IGNITION RESULTS

Figure 2 shows temperature profiles of Ti and Mg samples heated in O<sub>2</sub> at 1 atm. During the heating phase, all curves exhibit an increasing temperature with an intermediate plateau corresponding to the  $\alpha$ - $\beta$  solid-phase transition in Ti and the solid-liquid phase change in Mg. Occurrence of the critical temperature ( $T_{crit}$ ) marks the point at which heat generation from surface oxidation equals heat losses from the sample. A short time after  $T_{crit}$  is exceeded, a sudden change in the heating rate at the ignition temperature ( $T_{ign}$ ) coincides with the appearance of a luminous reaction front. The limit on the maximum operating temperature of thermocouples (2000 K) makes it impossible to measure combustion temperatures (above 3000 K).

In the case of Ti, a significant increase in the heating rate following the phase transformation near 1100 K signals the transition from a parabolic to a linear oxidation rate. Soon after, enhanced external oxidation produces rapid visible changes in surface texture and coloration, as seen in Fig. 3a. As  $T_{crit}$  is exceeded (around 1700 K), the metal specimen undergoes thermal runaway (even if the heat source is removed), leading to ignition on the upper surface at a few degrees below the melting temperature of Ti, 1933 K.

For Mg, the transition from a parabolic to a linear oxidation rate occurs before melting (around 800 K), as evidenced by the rapid formation of a white layer of magnesium oxide (MgO) around the outer surface of the sample (Fig. 4a). At normal gravity, the sample collapses after melting of the inner metal. The outer MgO layer remains in the solid phase and continues its growth as the temperature resumes its rise. Once  $T_{crit}$  is reached (near 1050 K), the oxide coating turns black and rapidly stretches in one direction until ignition occurs in the hottest spot (Fig. 4b). In the low gravity case, the specimen slowly grows upward, raising the high-temperature upper surface as a result of volumetric expansion after melting. As  $T_{crit}$  is exceeded, the top surface temperature rises exponentially as predicted by a kinetic-controlled oxidation rate. Vapor pressure increases and the sample rapidly stretches upward as the metal temperature approaches the boiling point of Mg (1380 K). Ignition occurs when enough Mg vapor reacts with oxygen to generate a visible flame on the sample top (Fig. 4b). In both the normal and low gravity cases, the thermocouple (not seen in Fig. 4) is located at the bottom of the sample to prevent its detachment from the continuously distorting upper surface. Therefore,  $T_{ign}$  shown in Fig. 2 is approximately 200 K below the actual  $T_{ign}$  (1300 K) in the top surface—as measured by a second thermocouple used in probing tests. The lag is caused by the rapid rise in temperature after  $T_{crit}$  compared to the longer diffusion time in the metal.

For both metals under normal and low gravity conditions, higher critical and ignition temperatures are obtained than the values reported in the literature [6,7]—more than 200 K above the highest measured temperatures for bulk metals. The disparity stems from the high heating rates applied in this investigation (above 100 K/s in the early stages), which are required for fast ignition during the low gravity time available in the aircraft. Although the applied heating rate is only 15% higher than the experimentally determined minimum ignition heat flux, it exceeds by two orders of magnitude the linear heating rates used for near-isothermal studies. The high heating rate applied to the top surface of the metal creates a significant temperature difference within the specimen (around 40 K in Mg and 150 K in Ti). In addition, the surrounding gas and vessel walls remain at a temperature close to ambient during the fast heating phase. As a result, higher  $T_{crit}$  and  $T_{ign}$  values are obtained as the large increase in internal conduction and radiation

heat losses overcomes the phase-change contributions to heat generation that generally determine ignition of Mg and Ti in isothermal experiments.

The apparent lack of influence of gravity on  $T_{crit}$  and  $T_{ign}$  is another consequence of the high value of conduction (for Mg) and radiation (for Ti) heat losses versus natural convection heat loss present at these temperatures. A general theoretical model was developed to calculate the effect of low gravity on  $T_{crit}$  for a metal sample subjected to conduction, convection and radiation heat losses, as well as heat generation by oxidation. For our experimental conditions, the model shows a reduction of approximately 10 K on  $T_{crit}$  at low gravity, a small value that is within experimental uncertainty.

## V. COMBUSTION RESULTS

### Titanium

After ignition at a point on the top outer rim of the sample, a molten mass consisting of a mixture of metal and oxides starts traveling across the upper surface of the specimen. Steady-state downward propagation of a spherical mass follows in a smooth, nonexplosive fashion until reaching the alumina base. The average steady-state propagation rates measured for the normal and low gravity cases are 16.2 mm/s and 8.7 mm/s, respectively. A steady regression behavior during this period at 1 g suggests that the propagation velocity has not been altered significantly by the influence of the gravity force on the molten mass—as confirmed by its spherical shape in Fig. 3b. Furthermore, a calculation of the Bond number ( $Bo = \text{gravitational force/surface tension force}$ ) under these conditions results in values below 0.1. Evaluating the ratio of propagation velocities at normal ( $V_n$ ) and low ( $V_l$ ) gravity gives a value of 1.86.

Following a theoretical approach similar to the one used for gaseous flame propagation, several studies [3,4] have obtained an expression of the form  $V \sim (w)^{1/2}$  to calculate the propagation velocity  $V$  along metal cylinders undergoing heterogeneous surface burning with a rate of reaction  $w$ . Considering the diffusion and convection of  $O_2$  to the sample as the rate-limiting step, the reaction rate becomes proportional to  $w \sim (Gr)^{1/4}$ , where  $Gr$  is the Grashof number. The propagation velocity then depends on  $Gr$  as  $V \sim (Gr)^{1/8}$ , so that  $V \sim (g)^{1/8}$ . Evaluating  $V_n/V_l$  for the normal (1 g) and low gravity ( $\pm 0.01$  g) cases, a theoretical ratio of 1.78 is obtained. The close agreement between experimental and theoretical ratios of propagation velocities indicates the importance of the influence of natural convection-enhanced oxygen transport on combustion rates.

The lower propagation rates observed at low gravity in this study differ from the higher regression rates in microgravity given by Steinberg et al. [1]. The disagreement arises from the different experimental configurations used. In Ref. 1, propagation rates are measured for cylindrical rods ignited at the bottom. The increase in regression rates in microgravity is attributed to an increase in the temperature of the retained molten mass; cyclical detachment of this mass occurs during upward propagation at 1 g. The influence of the gravity force on the molten mass (whose detachment subsequently reduces heat transfer to the unreacted metal) is apparently greater than the influence of buoyancy-induced convection on oxygen transport to the reaction zone. In the present investigation, only the latter influence is evaluated by comparing the propagation rate at low gravity with the 1 g steady-state downward propagation rate at low Bond numbers.

## Magnesium

Following the first flash generated by the Mg-O<sub>2</sub> homogeneous reaction, an ignition wave runs through the sample driven by the difference between the flame temperature (near 3430 K, the vaporization-decomposition point of MgO [8]) and the temperature of the unreacted metal, which is near the Mg boiling point. The average ignition wave speeds measured for the normal ( $V_n$ ) and low ( $V_l$ ) gravity cases are 220 mm/s and 115 mm/s, respectively. If the approximation used to compare propagating velocities in Ti rods is used to evaluate the  $V_n/V_l$  ratio of ignition wave speeds of Mg (similarly assuming a diffusion/convection controlled reaction rate), close agreement is again found between the experimental (1.91) and the theoretical (1.78) values. Owing to the irregularly shaped, porous oxide layer surrounding the sample—a consequence of metal melting before ignition—no attempt was made to calculate surface regression rates. Instead, an evaluation of burning times and a qualitative discussion of important phenomena is given.

From the visible images and emission spectra measurements, the structure of the luminous flame that engulfs the sample after the passage of the ignition wave is in general agreement with the extended reaction zone model from Glassman et al. [2]. An inner region of Mg-O<sub>2</sub> vapor-phase reaction is followed by MgO condensation in the bright white flame front observed in the photographs. However, a thin green emission band and a wider outer diffuse blue zone are visible in addition to the prescribed features from the model (Fig. 4c). This radiation may come from excited metal vapor created by oxide dissociation in the high-temperature front, by fine oxide particles, and possibly by Mg-O<sub>2</sub> heterogeneous reactions occurring in the oxide surface in a lower temperature region [9].

The spectroscopic measurements show similar behavior in the normal and low gravity cases. The familiar UV and green systems of MgO and Mg appear as the major radiation contributors. However, an interesting feature was captured by time-resolved spectroscopic measurements during the ignition wave propagation. Figure 6 shows the sequence of spectra for the UV and green systems from the onset of ignition to fully developed combustion in a low gravity experiment. The UV bands of MgO and the UV Mg triplets appear in emission during the early stages of the ignition process, followed by an absorption and emission equilibrium towards the end of the ignition phase. The line and band systems later exhibit reversal to absorption against an intense continuum background during fully developed combustion. In contrast, the green system of MgO and the green triplets of Mg appear in emission at all times. Although no conclusive explanation can be given at this time, it is believed that the reversal may be caused by the transition from chemiluminescence from early ignition reactions to thermal excitation of products in an extended reaction zone with background radiation from condensed oxides.

During fully developed combustion, buoyancy-generated convection currents are responsible for the main differences observed in the two cases investigated. At 1 g, high convection currents enhance burning by increasing O<sub>2</sub> flux to the reaction zone and by removing oxide products that may constitute a barrier to O<sub>2</sub> diffusion (Fig. 4c). The proximity of the resulting flame front to the metal sample is an indication of fast burning rates. In comparison, at low gravity conditions, the severe reduction of convection—threefold by the  $(Gr)^{1/4}$  dependence—and the increased resistance to O<sub>2</sub> diffusion by combustion products diminish the oxygen transport. Figure 4c shows a broader, particle-laden outer blue zone (undisrupted by convection currents) and a larger flame front detachment.



While in 1 g the products are swept upward by buoyancy-induced currents, condensed oxides rapidly accumulate and agglomerate in the reaction front at low gravity, producing a highly radiant flame front. Sporadic removal occurs only for large-diameter particles expelled by inertial forces and residual accelerations during the reduced-gravity trajectory (*g* jitter). Particle accumulation may account for the unique unsteady, spherically symmetric explosion phenomenon observed at low gravity—which we refer to as radiation-induced metal explosion (RIME). As shown in Fig. 5, a high particle density in the flame front generates a large heat flux to the sample. This effect raises the surface temperature and increases metal evaporation; the flexible oxide membrane that keeps Mg at temperatures below its boiling point expands as vapor pressure builds up inside the metal core. As evaporation increases, so does the flame front diameter to accommodate greater oxygen flux and maintain the stoichiometry. At the peak of the cycle, the amorphous specimen is transformed into a spherical core with twice the size of the original cylinder (Fig. 5b). Finally, the oxide layer explodes and breaks in multiple points (creating small jets), relieving its internal pressure and reducing its size while ejecting a large number of particles in the process (Fig. 5c). The RIME cycle may repeat several times during the burning stage. Although RIMEs are not observed at normal gravity, explosions and jets are still induced at 1 g by localized pressure bursts where the flame gets too close to the sample.

Burning times vary widely depending on the number of jets and explosions that accelerate combustion. Nevertheless, the average burning time at low gravity (4 s) is around twice the average value at 1 g. In some cases, longer burning times at low gravity may also result from the reduced reaction area produced by oxide walls surrounding the sample. These large oxide structures are built from condensed particles in the flame front using the alumina holder as an anchoring point (Fig. 4d).

## VL. FUTURE RESEARCH

Based on the results obtained to date, future experiments will explore the effects of pressure, O<sub>2</sub> concentration, and gravity level on the ignition and combustion of bulk metals. Higher pressures may enhance the role of convective heat losses at progressively higher gravity levels, so that ignition may occur at higher temperatures (and burning rates may increase) at normal and elevated gravity. In addition, the oxidation reaction may switch from being kinetic-controlled to a diffusion-controlled reaction in a low-O<sub>2</sub> concentration atmosphere; the enhanced transport of O<sub>2</sub> by convective currents may then increase the oxidation rate and cause ignition at lower temperatures under normal gravity conditions. Changes in the properties of the oxide layer formed before ignition under various O<sub>2</sub> concentrations may also affect the behavior of RIMEs.

Parallel to the experimental work, an analytical model of the ignition process is being developed to predict heating times, critical and ignition temperatures, and the relative magnitude of all the heat transfer mechanisms. The model may also help to determine oxidation rates and mechanisms in the high-temperature region close to ignition.

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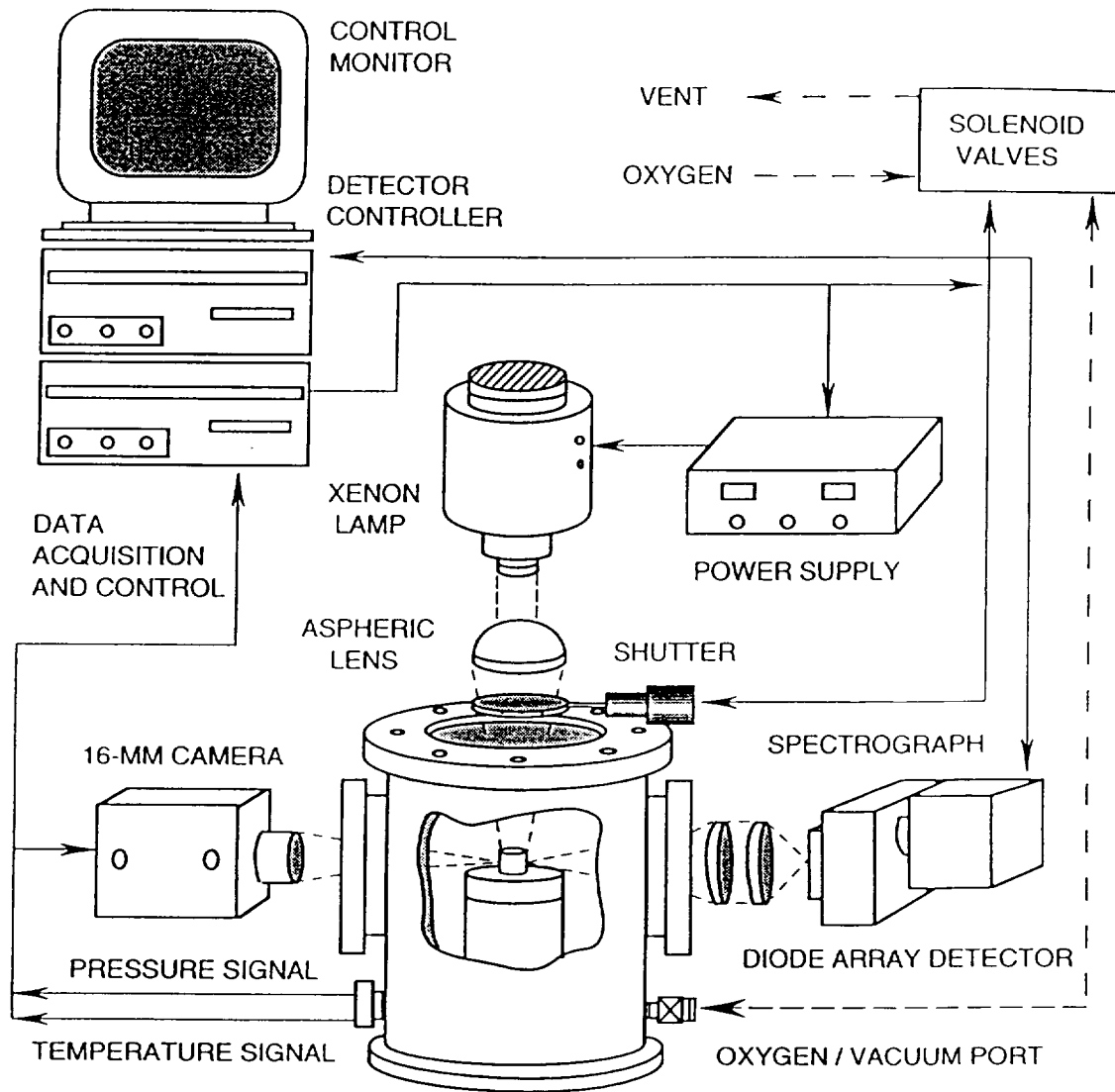
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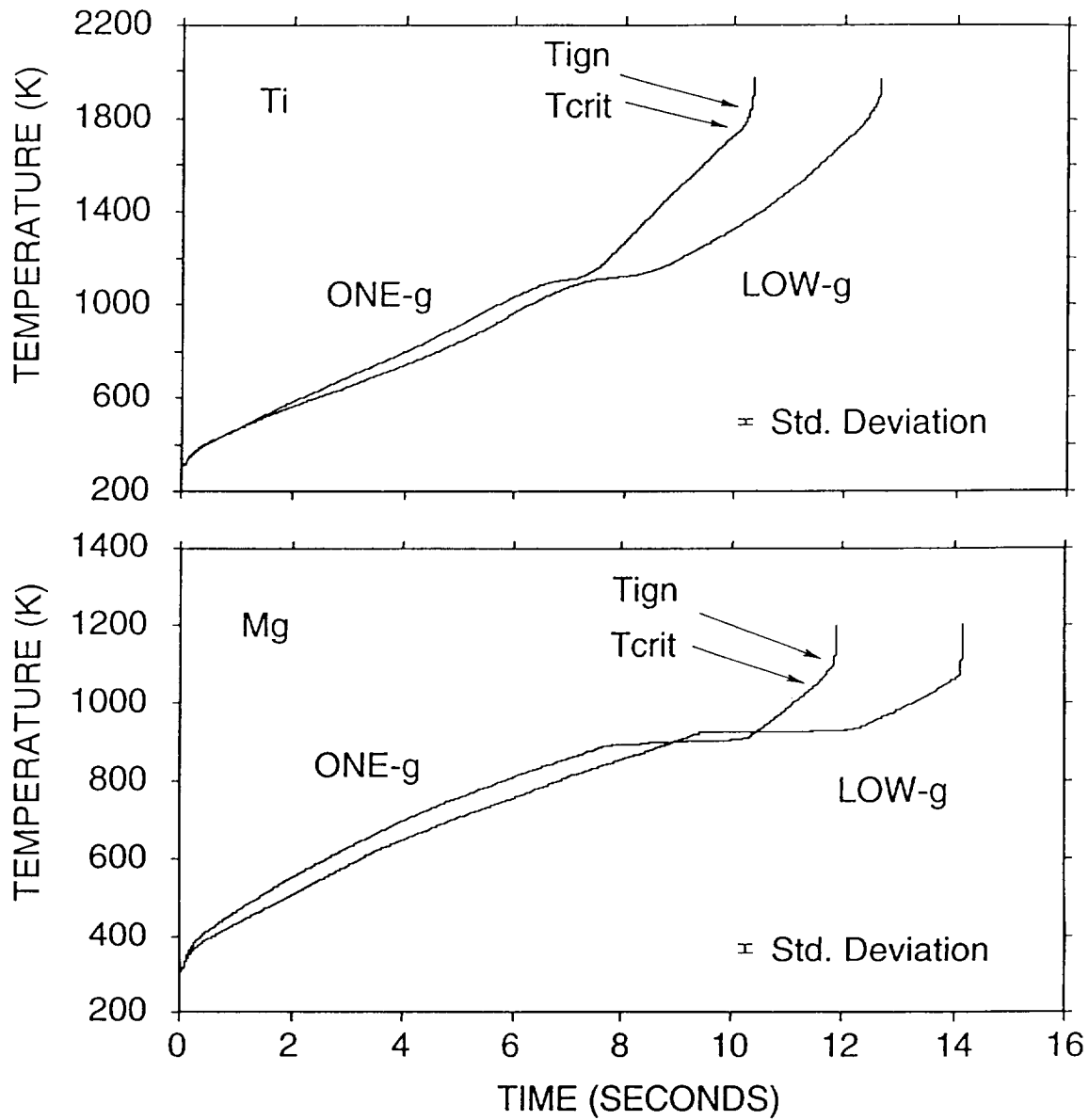
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#### **IX. PERSONNEL AFFILIATED WITH THIS RESEARCH**

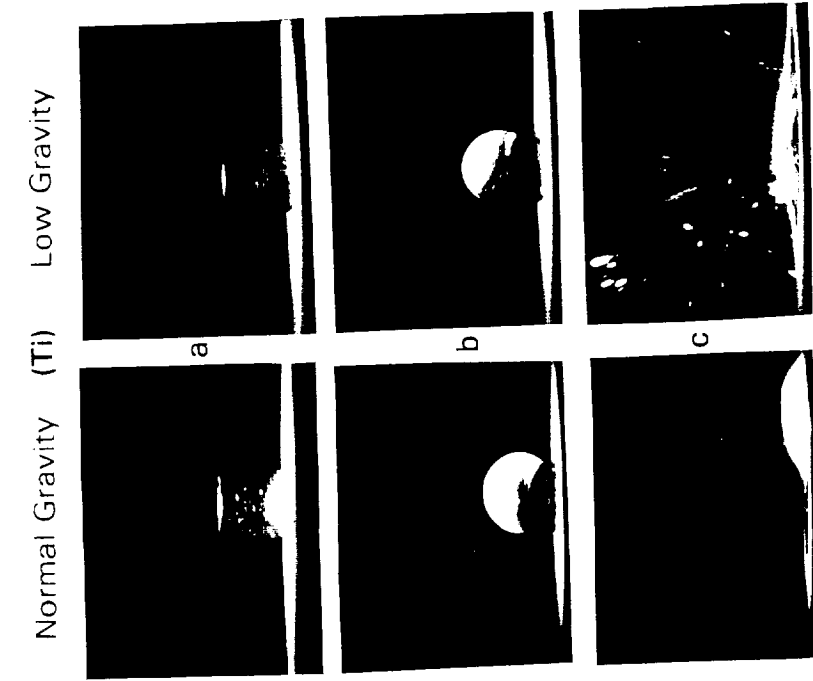
1. Melvyn C. Branch, Professor of Mechanical Engineering, Co-Principal Investigator.
2. John W. Daily, Professor of Mechanical Engineering, Co-Principal Investigator.
3. Angel Abbud-Madrid, Graduate Research Assistant, Ph.D. Candidate.
4. Christopher McKnight, M. S. Student.
5. Wesley T. Ramm, Undergraduate Student.
6. David T. Bunting, Undergraduate Student.



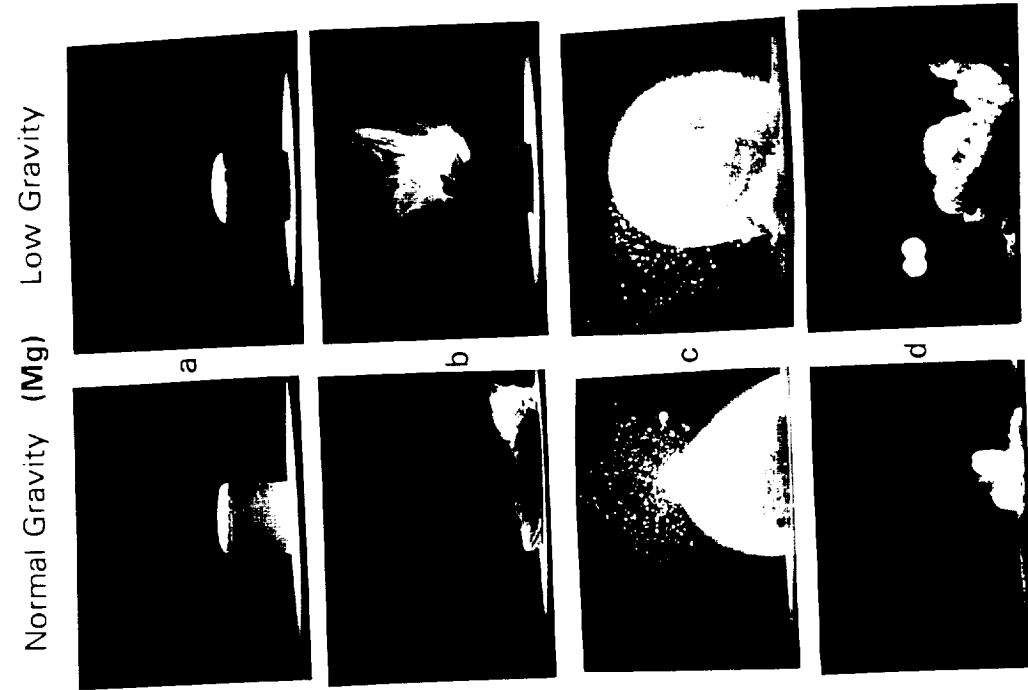
**Figure 1.** Schematic diagram of the experimental system.



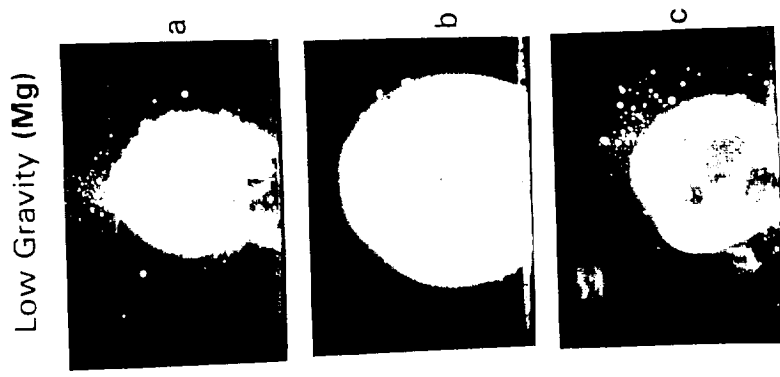
**Figure 2.** Surface temperature profiles of bulk Ti (top) and Mg (bottom) specimens in pure O<sub>2</sub> at 1 atm during the heating and ignition stages under normal and low gravity conditions.



**Figure 3.** Sequence of high-speed photographs of the heating, ignition and combustion events of bulk Ti specimens in pure  $O_2$  at 1 atm under normal and low gravity conditions. (a) Heating and surface oxidation (around 1400 K); (b) steady propagation (200 ms after ignition); (c) particle shower and fragmentation.

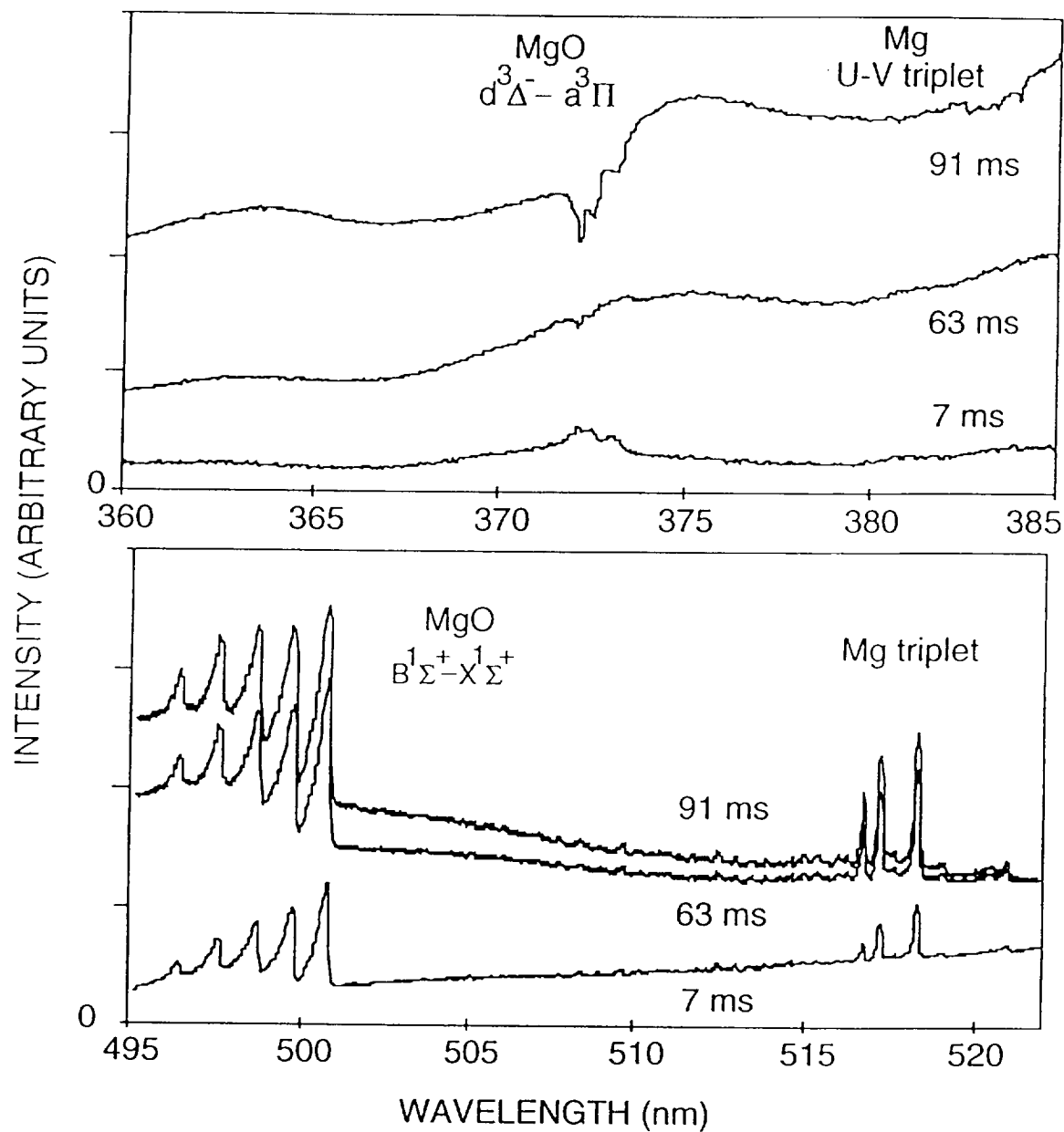


**Figure 4.** Sequence of high-speed photographs of the heating, ignition and combustion events of bulk Mg specimens in pure  $O_2$  at 1 atm under normal and low gravity conditions. (a) Beginning of melting stage (around 923 K); (b) ignition wave propagation (7 ms after ignition); (c) fully-developed combustion; (d) end of combustion.



**Figure 5.** Radiation-induced metal explosion (RIME) in a bulk Mg specimen at low gravity. (a) Start of cycle; (b) maximum flame diameter (c) oxide layer explosion.

5 mm



**Figure 6.** Time-resolved emission spectra from the UV (top) and green (bottom) systems of the Mg-O<sub>2</sub> gas-phase reaction at 1 atm in low gravity at 7 ms (start of ignition), 63 ms (end of ignition wave propagation), and 91 ms (fully developed combustion) after ignition.